

286870 52,492/64

COMMONWEALTH OF AUSTRALIA

PATENT SPECIFICATION

Class

Int CL

Application Number

52,492/64.

25.1.

C11d.

Lodged

4th December, 1964.

Accompanied by a

Provisional Specification.

Complete Specification

Entitled

FOAMING BACTFRICIDAL DETERGENT COMPOSITIONS

CONTAINING AN OXIDISING AGENT.

Lodged

10th November, 1965. 8th November, 1968.

Accepted **Published**

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Convention Priority

Applicant

IMPERIAL CHEMICAL INDUSTRIES OF AUSTRALIA

AND NEW ZEALAND LIMITED.

Actual Inventors

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Related Art:

49,236/64

25.1; 26.1.

270,424(12,196/61)

25.1; 87.1; 88.2.

262,897(18,403/62)

25.1; 88.2; 75.8; 28.8.

The following statement is a full description of this invention, including the best method of performing It known to US:

This inv ntion relat s to new compositions of matter and in particular to compositions of matter which contain a foam producing agent together with a stabiliser, a bactericide and an oxidising agent.

A common method of cleansing and sterilising milking machines and milk handling equipment comprises cleaning the equipment with one or more daily alkaline rinses followed by an occasional acid rinse, approximately one in every seven rinses, to remove the build-up of hardened milk proteins known as milk stone in milk carrying pipe-lines, holding vats, containers, tankers and like items of equipment. Even under optimum conditions milk stone build-up occurs and the plant must be dismantled andmetal parts which come into contact with milk must be cleanednechanically. In addition, sterilisation is normally achieved by passing scaling water through the equipment after cleansing, or by the use of chemical sterilisers. Heat sterilisation is difficult, since it involves the supply of boiling water or live steam, the temperature of which cannot be maintained through the length and breadth of the milk handling plant and since heat causes precipitation of milk proteins and consequent milk stone build-up. Chemical sterilisers used as rinses, on the other hand, provide only short time contact with metal surfaces and can be inefficient in providing a high level of bactericidal or bacteriostatic effect.

In our Patents Nos. 270424 and 262897 we have already disclosed that foaming bactericidal detergents based on a quaternary ammonium compound combined with a chemical steriliser provides a means of cold cleansing and sterilising of milk handling equipment with high bactericidal efficiency.

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While this treatment prevents milk stone build-up, it was found that under unfavourable conditions minute residues of the quaternary ammonium compound may still be left on the surfaces of metal and glass after rinsing with water and that these residues may suffice to inhibit the most sensitive of bactericidal cheese starters and thus may interfere with ordinary processes of cheese manufacture.

We have now found that this difficulty may be overcome by the use of new compositions of matter.

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Accordingly we provide new compositions of matter comprising firstly, as a forming agent, at least one quaternary armonium compound of the formula R1R2R3R4NX where R_1 is C_nH_2n+1 , n is 12 to 18 inclusive and X is chlorine, bromine or iodine and where R2 and R3, which may be the same or different, are an alkyl radical containing 1 to 3 carbon atoms and RA is an alkyl radical having 1 to 3 carbon atoms or a benzyl or B-phenoxyethyl radical and secondly, as a stabiliser, at least one substituted alkane of the general formula $C_m H_{2m+1} Y$, where m is 10 to 20 inclusive and where Y is -OH or NR₅R₆, where R₅ and R, which may be the same or different, are hydrogen or alkyl groups having from 1 to 3 carbon atoms inclusive and thirdly, at least one biologically acceptable oxidising agent. Suitable oxidising agents are the hypochlorites, perborates and peroxides and iodophor. By iodophor we mean a concentrated solution of iodine in a condensation product of an alkylphenol with ethylene oxide. The condensation product has an alkyl group with between 5 and 10 carbon atoms in the chain and comprises between 5 and 20 ethylene oxide units per phenol in the molecule. The most preferred condensation product is

the product available commercially under the registered trad mark "Lissapol" N; a suitable concentration of iodine in the condensate is 10% w/v of free iodine. bdophor concentrates can be made also from the polyoxyalkylene surfactants as disclosed in claim 1 of United States Patent No. 2,759,869and from mixtures of the latter with alkylphenol condensates as above described. It is also known that an acid environment is necessary to obtain the maximum biocidal efficiency of iodine. Therefore to prevent the decomposition of iodine, an iodophor is best sized with sufficient acid to maintain the desired degree of acidity in the water present at use dilution. Although may would are satisfactory, phosphoric acid is to be referred as in addition to its low toxicity and volatility, happing acid has a buffering action in the pH range of 3 alch is an excellent means of maintaining stability with the product. The preferred alkyl radicals in both a quaternary amnonium compound and the substituted is and defined above are the unbranched normal carbon ans.

Our compositions retain the detergent, foaming and brotesicidal properties of the quaternary anmonium compound the illiser mixtures; at the same time they have been form the effective in reducing build-up of "milk stone". In addition they reduce the concentration of the minute residues of the quaternary ammonium compounds on the surfaces of the equipment, e.g. metal and glass to such a level that the action of the most sensitive of the bacterial cheese starters is not affected and hence there is no interference with ordinary processes of chees manufacture.

Compounds of the tetraalkyl ammonium halide type have

bactericidal and bacteriostatic properties by themselves. However, under certain circumstances it may be desirable to intensify the bactericidal effect or to provide a broader spectrum offactivity; other compounds having bactericidal or bacteriostatic properties may then be added.

Accordingly we also provide a new composition of matter as defined above comprising in addition at least one bactericide and/or bacteriostat. One suitable bactericide is, for example, a guanidine salt of the general formula defined in claim 1 of Australian Patents Nos. 159, lll and 222,033. An example of this type of bactericide is bis-(p-chlorophenyl-diguanido)hexame.

Other suitable antibacterial and antiseptic compositions are the indole derivatives

Where R stands for hydrogen and wherein the nucleus

A may optionally bear one or more halogen atoms or nitro,

acylamino, alkyl hydroxy, alkoxy or carboxylic acid radicals.

These are defined and described in Australian Patent Specification

No. 221,724. An example of this type of bactericide is

3-(5'-nitro-2'-furfurylidene)oxindole.

The concentrations of surface active agent in the final liquid to be foamed which is required for high foam expansion ratios may vary over a wide range; the upper limit, apart from economic consid rations, is not critical.

All concentrations in this specification unless stated therwise ar given in percent w ight/v lume. Thus for example concentrations up to 20% and more of cetyl trimethyl ammonium bromide are feasible although in practice, because of cost, 1% would rarely be exceeded. The lowest useful concentration is about 0.05% of the liquid bulk. The preferred range for those uses where foam volume is more critical than extreme stability as e.g. for cleaning and disinfection is 0.1 to 0.6%.

The amount of stabiliser required is a function of the concentration of the surface active agent. Useful weight ratios of surface active agent to stabiliser range from 100:1 to 1½:1. Ratios of 50:1 are satisfactory for cleaning or disinfectant purposes; the preferred ratios for stable foams are 30:1 to 5:1. The concentration of the oxidant in the final liquid to be foamed is not narrowly dritical ranging from 0.001% to 0.2% of the liquid, the preferred range being 0.01% to 0.05%.

The concentrations of the bacteriostat in the final liquid to be foamed may also vary over a wide range and are known from the prior art. Suitable concentrations for treatment of milking equipment are e.g. from 0.0001% and even less to 0.02% of the liquid bulk but depend of course on the known potency of each bacteriostat.

The above concentrations refer to the final liquid to be foamed. For practical use it is desirable to prepare concentrates of the mixed ingredients containing less water. These concentrates may be diluted with water prior to foaming in a ratio ranging from 1:10 to 1:100. Aparticularly suitable ratio of dilution is 1:30.

The foaming efficiency as defined in our co-p nding

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application No. 12196/61 and f am stability ar excellent; the addition f the exident and/r the additional bacteriostats does not affect foaming properties.

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an inert gas into the aqueous solution, emulsion or dispersion of the compositions described above. The gas is usually air, but other inert gases such as nitrogen or carbon dioxide may be used. Means of introducing the gas into the liquid are known; vigorous agitation may be used, but the most practical method is introduction of a rapid stream of air into a stream of liquid with or without the use of special nozzles and distributors. One such method useful particularly for the practical use of our foaming compositions is described in our Australian Patent Specification No. 253,078; another is described by J.P. Fry and R.J. French in J. Application. Chem., 1st October, 1951, pp. 425-429. This latter method was used for the evaluation of the foams of the present invention.

A particularly useful application of our invention resides in the foam treatment of surfaces to be sterilised.

Thus we have found that the effective volume of water containing tetraalkyl ammonium halide and stabiliser and bacteriostat and an oxidant as described above may be increased by up to 60 times, over prolonged periods, when foams are prepared from the new compositions of our infention and applied to the surfaces to be treated.

Accordingly we also provide a method of cleaning and disinfecting surfaces comprising preparing an aqueous foam from the new compositions of the present invention and

applying the foam to the surface to be treat d. Highly spanded stabilised foams from the new compositions of the present invention can be used for cleaning and disinfecting agricultural equipment such as milking machines, milk, butter and cheese vessels, cream separators, bottling machines and generally in the manufacture of foodstuffs for the cleaning of plant. The commercial milking machine may be used without additional equipment, recirculating an aqueous solution of surfactant, stabiliser, oxidant and bacteriostat and bleeding sufficient air into the suction side of the machine to build up the foam. A liquid volume of only 1/10 to 1/60 of the total volume of the vessel and pipes is required to fill the vessels and pipes completely with foam.

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When used in food transport tankers such as milk tunkers, considerable saving not only in materials but in time required for disinfection may be made. In the conventional method of cleaning milk road transport tankers, it is necessary to fill the tanker with detergent solution, to flush the detergent out and then to spray sodium hypochlorite solution thoroughly on to all wall areas; recirculation of the sodium hypochlorite solution of the sodium hypochlorite solution and the time is necessary and it is difficult to ensure that all intricate areas are safely disinfected. A final flush with water is then required. With the compositions of this invention it suffices to fill one tenth of the tanker or less with the aqueous solution and to air blow briefly to produce the foan and then to rinse with water.

When cl aning industrial vessels it is sufficient to fill them with from 1/5 to 1/50 of their volume with water containing the new compositions of th present invention and

to blow inert gas into the solution. With simple, suitably designed nozzles, which are known p r se, particularly fine and stable foams can be made. Very substantial savings in the biologically active constituent can thus be achieved.

Furthermore we have found that if agricultural equipment is cleaned with the foaming compositions according to our applications 12196/61 and 18403/62 and if after the removal of the bulk of the expanded stabilised foam the equipment is washed separately and subsequently with an aqueous solution of an oxidant as defined above, then the concentration of the minute residues of the quaternary ammonium compound on the surfaces of metal and glass is reduced to such a level that the action of the most sensitive of the bacterial cheese starters is not affected.

Accordingly we also provide a process which comprises exposing the surfaces of equipment with foaming compositions according to our applications 12196/61 and 18403/62, removing said foaming compositions from said equipment and, subsequently, treating said equipment with aqueous solutions of an oxidant as defined above. The concentration of the oxidant expressed as mole percent of the aqueous solution in the aqueous wash solution is not narrowly critical; it ranges from 0.003% to 0.07% of the liquid bulk, the preferred range being 0.01 to 0.04%.

Our invention is now illustrated by but not limited to the following examples.

Example 1

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A mixture comprising 10g of solid cetyl trimethylammonium bromide, 3 g of solid cetyl alcohol and 0.15 g of chlorohexidine digluconat was added to 80 ml of hot water and thoroughly

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mix d. Aft r the mixture had c ol d to ab ut 20 C,
0.6 ml of an aqueous solution f sodium hyp chlorit
(10% w/v av. chlorine) was added while stirring and the
whole volume adjusted to 107 ml. The mixture so formed
was diluted further by adding to 1 part of the mixture
an additional 30 parts of water and mixing thoroughly.
There was thus obtained a bactericidal foaming composition
suitable for the cleaning of milking machines and not
detrimental to cheese starters.

30 fluid oz of this dilutedmixture was introduced into a typical commercially available four imit milking machine and using the air-bleed technique previously described a stable foam was formed which filled all the interior of the milking madhine. The foam so formed was left in situ for six hours and then flushed from the machine by pumping through its interior 4 gallons of water. Ten gallons of milk from which a control sample had been taken to represent the bulk of themilk, were then passed through the machine as would occur in the normal milking process. Samples were taken from the first gallon leaving the machine, from the fifth gallon leaving the machine and from the bulked milk into after passage through the machine. The samples referred to above were then submitted to tests which determined whether the samples inhibited the efficiency of cheese starters. The results are shown in Table I.

*Cheese Starter No. C.13 Samples of Milk	% of Inhibition of Cheese Starter
Control	Nil
lst Gallon	Insignificant
5th Gallon	Nil
Bulked in toto	N1l

*Cheese starter culture Str. cremoris C113

Example 2

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A mixture comprising 10 g of solid cetyl trimethylammonium bromide, 3 g of solid cetyl alcohol and 0.15 g of chlorohexidine digluconate was added to 80 ml of hot water, thoroughly mixed and the volume adjusted, after cooling to about 20°C, to 100 ml.

The mixture so formed was diluted further by adding to 1 part of the mixture an additional 30 parts of water and mixing thoroughly by stirring.

above was introduced into a typical commercially available four unit milking machine and, using the technique of air-bleed previously described, a stable four was formed which filled the interior of the nilking machine. The foam so formed was left in situ for six hours and then flushed from the machine by pumping through the machine 4 gallons of water. 50 gallons of milk, from which a control sample had been taken to represent the bulk of the milk, was then passed through the machine, as would occur in the normal milking process, and typical samples were taken from the first gallon leaving the machine, the fifth gallon leaving the machine,

in toto after passage through the machin. The samples referred to above were then submitted to tests which showed whether the samples inhibited the efficiency of cheese starters. Results are shown in Table II.

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Table II

	% Inhibition of	Cheese Starters
Sample of Milk	*Cheese Starter No. C.11	Cheese Starter No. C.13
Control	Nil	D11
lat Gallon	50	94
5th Gallon	25	88
Oth Gallon	19	79
1 od n toto	18	78

starter culture Str. cremoris

This example illustrates that cheese starters are inhibited by milk which has been passed through a milking machine treated with a cleaning mixture in the absence of an oxidant.

Example 3

Example 2 was repeated, except that instead of pumping 4 gallons of water through the machine to flush the foam from the interior, 4 gallons of water containing 200 parts per million of sodium hypochlorite were pumped through the machine to flush the foam from the interior of the machine.

Table III

Sample	% Inhibition of Che	ese Starters
of Milk	*Cheese Starter No. C.11	*Cheese Starter No. C.13
Control	Nil	Nil
lst Gallon	Insignificant	Insignificant
5th Gallon	N11	N11
50th Gallon	Nil	Nil
Bulked in toto	Nil .	Nil

* Cheese starter culture Str. cremoris

This example illustrates that cheese starters are not inhibited by milk whichhas been passed through a milking machine subjected to the after-treatment with an oxidant.

Examples 4 to 15

Example 1 was repeated but instead of using 10 g of solid cetyl trimethylammonium bromide, 3 g of solid cetyl alcohol, 0.15 g of chlorohexidine digluconate, 0.6 ml of sodium hypochlorite dissolved in 100 ml of water and finally diluting 1 part of this mixture with an additional 30 parts of water, aqueous mixtures were prepared using the appropriate concentrations list d in Table IV.

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There were thus obtained fouring compositions and the like.

Example 16

Example 1 was repeated but the 0.5 ml of sodium hypochlorite was replaced by 3 ml of iodophor containing 10% w/v of free iodine. There was thus obtained a foaming composition suitable for the cleaning of milking machines and the like.

Example 16

Example 1 was r peated but the 0.6 ml of sodium hypochlorite was replaced by 3 ml of iodophor containing 10% w/v of free iodine. There was thus obtained a foaming composition suitable for the cleaning of milking machines and the like.

Example 17.

Example 1 was repeated but the 0.6 ml of sodium hypochlorite was replaced by 1.5 ml of iodophor containing 10% w/v of free iodine. There was thus obtained a foaming composition suitable for the cleaning of milking machines and the like.

Example 18

Example 2 was repeated, except that instead of pumping 4 gallons of water through the machine to flush the foan from the interior, 4 gallons of water containing 1000 parts per million of iodophor containing 10% w/v of free iodine were pumped through the machine to flush the foam from the interior of the machine. After this treatment no inhibition of cheese starter cultures Str. cremoris
C.ll and C.l3 was found. This example illustrates that cheese starters are not inhibited by milk which has been passed through a milking machine which has been subjected to the after-treatment with an oxidant.

Example 19

Example 1 was repeated but the 0.15 g of chlorohexidine digluconate was replaced by 0.0000075 g of 3(5'-nitro-2'-furfurylidene) oxindole. There was thus
obtained a bactericidal foaming composition suitabl for
the cleaning of milking machines and not d trimetal to chees
starters.

Example 20

Exemple 1 was rep ated, but the 0.15 g of chlorohexidine

digluconate was replaced by 0.0006 g of 3-(5'-nitro-2'-furfurylidene) oxindole. Ther was thus obtained a bactericidal foaming composition suitable for the cleaning of milking machines and not detrimental to cheese starters.

Example 21

Example 1 was repeated but the 0.15 g of chlorohexidine digluconate was replaced by 0.00006 g of 3-(5'-nitro-2'-furfurylidene)oxindole and the 0.6 ml of sodium hypochlorite was replaced by 0.3 ml of iodophor containing 10% w/v of free iodine. There was thus obtained a forming composition suitable for the cleaning of milking machines and not detrimental to cheese starters.

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18	12		10	, 9	æ	7	5	ს	4		Example No.
$R_1 = Stearyl$ $R_2 = R_3 = R_4 = Methyl$	$R_1 = \frac{My}{1}$ ristyl and Cetyl $R_2 = R_3 = R_4 = Methyl$	$R_1 = Wyr1$ etyl and Cetyl $R_2 = R_3 = R_4 = Methyl$	Rl = Myrietyl R2 = Benzyl, R3=R4=Methyl	R ₁ = Myristyl R ₂ =R ₃ =R ₄ = Methyl	R ₁ = Myristyl R ₂ =R ₃ =R ₄ =Wethyl	R ₁ = Myristyl R ₂ =R ₃ =R ₄ = Methyl	R1 = Myrietyl R2=R3 =R4 = Methyl	R1 = Cetyl R2=R3=R4 = Nothyl	R1 = Cetyl R2=R3=R4 = Methyl	Compound	Quaternary foaming agent
2	w	7	5	3	3	w	5	3	w	Cono. g/1	R ₁ R ₂ R ₃ R ₄ IIX
	₹ 1 a 1	2 = 00:71	R = Myrintyl Y = OH	R = Lauryl Y = LH ₂	R = Myriotyl Y = CH	R = Lauryl Y = OH	R = Myristyl Y = Oil	R = Cotyl Y = OH	R = Myristyl	Compound Co	Stabiliner
		0.35	0.5	0.3	0.3	0.5	0.5	0.7	Ö Ü	onc. E/1	r 27
İ		۶۰	> -	>-		: <u>.</u>	es	>-	}-	Compound	Jactericide
9)	ند ي	. 50	50	50	100	∾	2	50	50	Conc. p.p.m.	[.] •
2 (2) 2 (2) 12		Sodium Pertorata	Ивост	Iodophor	Iodophor	Naccl	Nacci	MACCI	Tocon	Compound	Oxident
50	100	000	200	100	50 6	200	500	000	200	Conc.p.p.m	23 6 +

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1 0.3 h	NO.	Quaternary foaming agent R ₁ R ₂ R ₃ R ₄ NX Compound Cono. g/1	nt R ₁ R ₂ R ₃ R ₄ NX Cono. g/1	Stabil	Stabilisor RY bund Cono. g/1		olde or a
R1 = Stearyl R2=R3=R4 = Methyl 3		e o mp o tasta	Cono. 8/1	Compound	Cono. g/l	Compound	Conc. p.p.m. Compound
R = Myristyl and Cetyl R = Myristyl O.1 A	14	R = Stearyl R2=R3=R4 = Methyl	ω _.	R = Stearyl	0.3	٨	50
	15	R1 = Myristyl and Cetyl R2=R3=R4 =Methyl	μ .	R = Myristyl Y = OH	0.1	Α	100

-chiorphenyldiguanido)hexane,

Compound B is 3-(5(-nitro-2'-furfurylidene) oxindole,

X = Bromine

286870 Table IV Continued

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The claims defining the invention ar as follows:-

- 1. Comp sitions of matter comprising firstly, as a foaming ag nt, at least one quaternary ammonium compound of the formula $R_1R_2R_3R_4NX$ where R_1 is C_nR_{2n+1} , n is 12 to 18 inclusive and X is chlorine, bromine or iodine and where R_2 and R_3 , which may be the same or different, are an alkyl radical containing 1 to 3 carbon atoms and R_4 is an alkyl radical having 1 to 3 carbon atoms or a benzyl or β -phenoxyethyl radical and secondly, as a stabiliser, at least one substituted alkane of the general formula $C_mR_{2m+1}Y$, wherein m is 10 to 20 inclusive, Y is -OH or NR_5R_6 and R_5 and R_6 , which may be the same or different, are hydrogen or alkyl groups having from 1 to 3 carbon atoms inclusive and thirdly, at least one biologically acceptable oxidising agent. (4th December, 1964)
- Compositions of matter according to claim 1 wherein the alkyl modicals $C_n H_{2n+1}$ in the quaternary armonic compound wi the likyl modicals $C_n H_{2n+1}$ in the substituted accuse are unbranched normal combon chains. (4th December, 1964)
- 3. Compositions of watter decording to chain 1, wherein the stellinger is one of and wherein $m=n^{\frac{1}{2}}$. (4th Lecember, 1974)
- 6. Compositions of matter according to chaim 2, wherein the attabilizer is an a line and wherein the z=2. (4th December, 1964)
- 5. Compositions of matter according to any one of the proceding claims wherein n is between 14 and 16 inclusive. (4th December, 1964)
- 6. Compositions of matter according to claims 1, 2, 3 and 5 wherein the stabiliser is tetradecyl alcohol. (4th December, 1964)

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- 7. Compositions f matter according to claims 1, 2, 3 and 5 wherein the stabiliser is cetyl alcohol. (4th December, 1964)
- 8. Compositions f matter according to claim 7 wherein the cetyl alcohol is commercially available cetyl alcohol.

 (4th December, 1964)
- 9. Compositions of matter according to any one of the preceding claims wherein the biologically acceptable omillaing agent is the salt of an alkali retal or alkaline earth sold and hypochlorous acid. (tth December, 1964)

 10. Compositions of matter according to claim 3 who wing the solution present in an amount between 50 and 250 control of available colorine per million

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 - 1... Pulpositions of matter surviving to suggeste of the largest state of the largest related son fitting to suggest the largest state of the largest related to the largest residue. (The region, 1964)

lesponsions of matter acquelling to the 15 wherein the

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bactericid is a guanidine salt of the general formula as defined in claim 1 or Australian Patent Specifications Nos. 159111 and 222033 (4th December, 1964)

- 17. Compositions of matter according to claim 15 wherein the bactericide is bis-(p -chlorophenyldiguanido)benzene.

 (4th December, 1964)
- 18. Compositions of matter according to claim 15 wherein the bactericide is an indole derivative of the general formula as defined inclaim 1 of Australian Patent Specification To. 221724. (4th December, 1964)
- Compositions of matter according to claim 15 wherein a pactericide is 3-(5'-nitro-2'-furfurylidene) exindole.
 - Tompositions of matter according to any one of the constitution of states and sherein to many ammonium compound is present in an amount to 4 and 15 by seight of the total composition.

in collions of witter locarding to claim 20 wherein to chair 20 wherein to chair amount in the chair and amount and all fault 0. The couplet of the total emporition. (the colline 1914)

intions of culter absorbing to any one of the course with the following simultaneous present in a course with the sense 1:100 and 1:12 of the quaternary composed. (4th Telegaper, 1964)

in the state of th

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 between 0.001% and 0.2% by weight of the total composition.
 (4th December, 1964)
- 25. Compositions of matter acc rding to claim 24 wherein the oxidant is present in an amount between 0.01% and 0.05% by weight of the total composition. (4th December, 1964)
- 26. Compositions of matter according to any one of the preceding claims wherein the bactericide is present in an amount between 0.001% and 0.02% by weight of the total composition. (4th December, 1964)
- 27. Compositions of matter substantially as herein described with reference to any one of Examples 1, and 3 to 15 inclusive (4th December, 1964)
- 28. A process of cleaning and disinfecting surfaces using compositions of matter according to any one of the proceding claims. (4th December, 1964)
- A process for exposing the surfaces of equipment with fouring compositions as defined in Amstralian Patents 7 as 2011; and 201097, recoving said fouring acaptaitions for a solice surposed and, or requestly, treating said equipment of courses solice as of an action on a consolar defined.

 (5): Desember, 1964)
- policy of retter invertee; to claim 14 wherein the attitude in present in an arount between 60 and 100 parts by weight of available rodine per million parts by weight of the total composition. (10th November, 1965)
- Sile Compositions of matter according to claims 18 and 19 Compositions of matter according to claims 18 and 19 Composition 3-(5'-mitro-2'-furfurylidene)oximiole is present in more until theeve 0.0005 and 0.00 parts of million 19 (30) Towarder, 190)

inclusive. (10th November, 1965)

DAT ID this 8th day of Hovember, 1965.

IMPERIAL CHEMICAL INDUSTRIES OF AUSTRALIA AND HEF ZEALAND LIMITED